

Standard Operating Procedure for the synthesis of Mesoporous Silica Nanoparticles (MSN):

Materials:

1.0 g cetyltrimethylammonium bromide (CTAB)

480 ml nanopure water

3.5 ml 2M sodium hydroxide (NaOH)

5.0 ml tetraethyl orthosilicate (TEOS)

Procedure:

In a fume hood a 1000 ml round bottom flask is charged with 1.0 g CTAB, 480 ml nanopure water, 3.5 ml 2M NaOH, and a magnetic stir bar. This was heated to 80 °C in a fume hood. Once at 80 °C 5.0 ml TEOS is added dropwise via syringe. The white precipitate forms after addition of ~3 ml TEOS. This white suspension is vigorously stirred at 80 °C for 2 hours. After stirring for 2 hours the precipitate is filtered in a fritted glass funnel (fine porosity) in a fume hood. The precipitate is washed with copious amounts of water and methanol. The MSN should be left under vacuum until completely dried in the fume hood. After transferring the MSN to a sample vial the MSN are further dried under high vacuum at 50 - 80 °C or freeze dried overnight.

Surfactant Removal:

The surfactant is removed by either an acid extraction or calcination. For calcinations either a tube or chamber furnace placed in a fume hood is employed; 1.0 g as-syn MSN is heated to 600 °C for six hours at a ramp rate of 2.0°/min. For acid extraction, 1.0 g as-syn MSN is suspended in 100 ml of methanol in a fume hood and 1.0 ml concentrated HCl is added. This is heated to 60 °C for six hours while vigorously stirring. The surfactant-removed MSN is filtered in a fritted glass funnel and washed with methanol and water in a fume hood.

Users:

Understand that this is a research laboratory and this synthesis is a common one. Organosilane functional groups may need to be added as needed.

Prof. Victor S.-Y. Lin Brian Trewyn Jennifer Nieweg **Igor Slowing** Cedric Po-Wen Chung Carla Kern Tae-Wan Kim Supratim Giri Tommy Hung-Ting Chen Yang Cai Juan Luis Vivero Nikola Knezevic Yulin Huang Chip Peeraphatdit Wei Huang Dorothy I-Ju Fang Cristina Bonaccorsi Robert Roggers Kevin Wu Yannan Zhao Steven Chih-Hsiang Tsai Michael Nahella



Standard Operating Procedure for working and experimenting with mesoporous silica nanoparticles (MSN):

A. Dry MSN in the powder form:

- 1. Whenever possible dry MSN should be worked with inside a fume hood. This includes loading sample vials, loading nitrogen sorption sample tubes for measurement of surface area and pore size, etc.
- 2. The dry atmosphere in the winter months cause static electricity to build up on the surface of the MSN and aeration of the particles occurs more frequently. Special care must be taken as needed, this includes using a fume hood and wearing a dust mask when working with the MSN.
- 3. After synthesizing the MSN they must be filtered to remove them from the aqueous suspension. This is accomplished by filtering in a fritted glass funnel attached either to a water aspirator or a vacuum pump. The end product is dry MSN so this filtering should be done in a fume hood.
- 4. When weighing MSN sample at the scale, only scales with protective shields should be used and MSN should only be manipulated and removed from sample vials inside the scale. (opposite side of shield as user)
- 5. The MSN are silicon dioxide (SiO₂) and are biocompatible of biological systems studied to date (this includes research done in this laboratory); however, it is good practice to wash hands following contact with MSN.
- 6. Goggles and gloves must be worn whenever using MSN, to avoid contact of MSN with eyes and skin.

Prof. Victor S.-Y. Lin Brian Trewyn Jennifer Nieweg **Igor Slowing** Cedric Po-Wen Chung Carla Kern Tae-Wan Kim Supratim Giri Tommy Hung-Ting Chen Yang Cai Juan Luis Vivero Nikola Knezevic Yulin Huang Chip Peeraphatdit Wei Huang Dorothy I-Ju Fang Cristina Bonaccorsi Robert Roggers Kevin Wu Yannan Zhao Steven Chih-Hsiang Tsai Michael Natella